Technical Paper 368

DEPARTMENT OF COMMERCE
HERBERT HOOVER, SECRETARY

BUREAU OF MINES
D. A. LYON, ACTING DIRECTOR

PARAFFIN WAX

AND ITS PROPERTIES; METHODS OF TESTING WAX AND OF ANALYZING OIL-WAX MIXTURES

BY

L. D. WYANT and L. G. MARSH

This paper represents work done under a cooperative agreement
between the Bureau of Mines, Department of Commerce
and the State of Oklahoma

PRICE, 10 CENTS
Sold only by the Superintendent of Documents, Government Printing Office
Washington, D. C.

WASHINGTON
GOVERNMENT PRINTING OFFICE
1925

U.S. Govt.
9r
10-19-1925

CONTENTS

Page.
Introduction — 1
  Scope of report — 2
Acknowledgments — 2
Paraffin wax — 2
  Characteristics — 2
  Occurrence — 3
  Manufacture — 3
Testing methods in present use — 4
  Oil content — 4
  Melting point — 5
    The A.S.T.M. paraffin-wax melting-point method — 5
    The A.S.T.M. petrolatum melting-point method — 6
    The value of melting-point determination of oil-wax mixtures — 6
  Specific gravity — 8
  Water content — 8
Solubility of paraffin wax and oils in acetone — 8
  Characteristics of an ideal solvent for paraffin oil-wax mixtures — 9
  Solvents studied — 9
  Advantages of acetone — 10
  Quality of acetone used — 10
  Preparation of oil-free paraffin waxes — 11
  Method of determining solubility in acetone — 12
  Effect of wax concentration on solubility — 14
  Relation between melting points and solubility — 14
  Preparation of wax-free oils — 14
Method of determining the paraffin-wax content of oil-wax mixtures — 17
  Intersolubility of oil and wax in acetone — 17
  Wax concentration — 18
    Method 1 — 18
    Method 2 — 18
    Method 3 — 19
    Application of methods — 19
  Temperature of filtration — 20
  Standardizing equipment — 21
  Testing for wax content of oil-wax mixtures — 21
    Sampling of oil-wax mixtures — 21
    Procedure in tests — 22
  Application of method — 23
Summary — 24

ILLUSTRATIONS

Page
PLATE I. *A*, Wax-distillate chilling machine; *B*, Interior of a pressroom, showing wax press and room-cooling coils 4
II. *A*, Wax-sweating house, showing interior arrangement of sweating pans; *B*, *a*, Saybolt improved wax tester; *b*, A. S. T. M. paraffin-wax melting-point apparatus assembly; *C*, A. S. T. M. petrolatum melting-point apparatus arranged to run duplicate tests 5
III. Wax storage and barreling house, containing barreled wax ready for shipment 4
IV. Assembly of constant-temperature filter-bath arrangement used in separating oil-wax mixtures with acetone: *A*, Top view; *B*, side view 5
FIGURE 1. Typical cooling curves of four typical waxes 7
2. Effect of crystallization from acetone on the melting point of a typical paraffin wax 12
3. Detail of set-up of U tube and sample bottle used in making solubility determinations 15
4. Solubility curves of three oil-free waxes 16
5. Solubility curves of four wax-free oils 17
6. Filtering bath and filters 21

Original from UNIVERSITY OF MICHIGAN

PARAFFIN WAX AND ITS PROPERTIES; METHODS OF TESTING WAX AND OF ANALYZING OIL-WAX MIXTURES

By L. D. Wyant and L. G. Marsh

INTRODUCTION

Lubricating oil is one of the most important and essential products of petroleum, as evidenced by the widespread use of petroleum lubricants in factories, machine shops, and powerhouses, and particularly in motor cars and motor trucks. The total production of lubricating oil in the United States during 1923, very nearly 1,100,000,000 gallons,[1] shows how widespread is the use of this commodity.

Lubricating oil of high quality can be made from almost any crude petroleum but because of differences in their chemical nature it is very much easier to make lubricating oils from some crudes than from others. To find out in detail why this is so the Bureau of Mines is making an intensive study of the whole subject of lubricating-oil and paraffin-wax manufacture.

At least 75 per cent of the lubricating oil manufactured in the United States is made from crude oil that contains paraffin wax (so-called "paraffin base" or "intermediate base" crude). This wax has a boiling range similar to that of the lubricants found in the crude, consequently the lubricating distillates obtained from these crudes contain wax that must be removed before satisfactory lubricants can be produced. The magnitude of the problem becomes evident when statistics are studied. The amount of lubricating oil and paraffin wax produced in the United States in the last six years is listed in Table 1.[2]

Table 1.—*Production of lubricating oil and paraffin wax, 1918–1923*

Year	Lubricating oil	Paraffin wax
	Gallons	*Pounds*
1918	841, 465, 767	505, 144, 357
1919	846, 760, 017	467, 235, 223
1920	1, 046, 708, 349	541, 404, 537
1921	877, 859, 074	433, 887, 214
1922	978, 752, 469	462, 003, 179
1923	1, 097, 368, 222	466, 646, 551
Total	5, 687, 913, 898	2, 876, 321, 061

---

[1] Hill, W. C., Petroleum refinery statistics for 1923: Bureau of Mines, 1924, p. 31.

[2] Hill, W. C., work cited, pp. 29–31.

Separation of wax from oil is often a very difficult and expensive process; in fact, many progressive refiners consider wax a necessary evil. The price received for finished paraffin wax is frequently less than the total cost of recovering and refining it. However, the expense of separating crude wax from the lubricating oil is a large part of the total cost, so that the actual refining of wax is usually profitable.

SCOPE OF REPORT

A preliminary survey of the problem of manufacturing lubricating oils shows that the separation of oil and wax is extremely important. Before this problem could be investigated, however, it was found necessary to study in detail methods for measuring the concentration of paraffin wax in oil-wax mixtures and to devise new methods which would be more satisfactory for research than any in present use. This report covers the work that has been done along this line. The present methods of testing oil-wax mixtures and commercial paraffin waxes are compared and a new method that depends upon the relative insolubility of paraffin wax in acetone is described.

ACKNOWLEDGMENTS

This investigation was made under the supervision of F. B. Tough, former chief petroleum engineer, and H. H. Hill, chief petroleum engineer, of the Bureau of Mines. The authors wish to thank N. A. C. Smith, petroleum chemist of the bureau, for the many helpful suggestions made during the progress of this report, and T. E. Swigart and M. J. Kirwan, former superintendents of the Bartlesville station of the bureau, for helpful suggestions and constructive criticism given while this paper was being prepared for publication. Special acknowledgements are extended to Mr. H. T. Bennett, chief research chemist, and Mr. E. W. Espy, chief experimental chemist, of Cosden & Co., and to Mr. A. Henricksen, chief chemist of the Marland Refining Co., for their assistance in compiling some of the data presented. Miss Ruth Brown of the Bartlesville station prepared the charts and drawings. The courtesy and assistance given by many refining companies who supplied the samples used in this work are sincerely appreciated.

PARAFFIN WAX

CHARACTERISTICS

The term "paraffin wax" as used in this report refers only to paraffin wax obtained in the commercial refining of crude petroleum. Although the pure paraffin wax as obtained from this source is un-

Digitized by Google Original from UNIVERSITY OF MICHIGAN

doubtedly a mixture of various paraffin hydrocarbons, nevertheless, it may be described as follows:

Paraffin wax when melted is colorless, odorless, and tasteless. When cooled below its melting point it is a translucent, apparently amorphous solid. Pure waxes with melting points as high as 170° F. are reported[3] as occurring as constituents of petroleum but the paraffin waxes of commerce, as generally produced in the refining of petroleum, have melting points between the limits of 115° and 150° F., usually between 120° and 130° F. Air mixed into cooling paraffin wax gives a snowy white opaque appearance to the solidified mass.

The high melting point of paraffin wax, and the facts that the wax does not deteriorate, is impervious to water, and is an excellent nonconductor of electricity, give it a very extended range of uses.

OCCURRENCE

Pure paraffin wax is seldom if ever found in nature, but has been isolated from a large number of widely distributed substances.[4] Commercial paraffin wax is derived mainly from petroleum and to a lesser extent from shale oil, ozokerite and coal, and lignite distillates.

MANUFACTURE

The commercial refining of petroleum is a complicated process[5] requiring especially designed and expensive equipment and many intricate and detailed methods. The crude oil is first separated by distillation into fractions generally known as "crude naphtha," "kerosene distillate," "gas oil," "paraffin distillate," and "residuum." Each of these fractions may be refined into various products by further fractionation and special processing.

The major part of the fraction called paraffin distillate is lubricating oil in which is dissolved the paraffin wax subsequently recovered as commercial paraffin.

Paraffin distillate is divided by chilling and pressing into two products, crude paraffin wax (called "slack wax") and oil (called "pressed distillate"). This pressed distillate is the material from which most petroleum lubricants are made. The separation by pressing is never complete and after the pressing the oil contains small amounts of dissolved wax, and the slack wax contains 30 to

---

[3] Mabery, C. F., On the composition of petroleum; the solid paraffin hydrocarbons that collect in oil wells in Pennsylvania: Am. Chem. Jour., vol. 33, 1905, p. 285.

[4] Brooks, Benjamin T., The nonbenzenoid hydrocarbons. Chemical Catalog Co., Inc., 1st ed., 1922, p. 95.

[5] Smith, A. D., Refining; Chapter XI of "A handbook of the petroleum industry," David T. Day, editor in chief. Vol. 2, 1922, pp. 322–420. Bacon, R. F., and Hamor, W. A., The American petroleum industry. Vol. 2, 1916, pp. 487–510.

60 per cent of oil and low-melting paraffin. Plate I, *A*, is a wax-distillate chilling machine and Plate I, *B*, is the interior of a press-room showing a wax press and cooling coils.

Slack wax is refined to commercial paraffin by "sweating", a process of slow fractionation that depends on the difference in melting points of the oils and waxes present. In general the practice of sweating[6] embraces the following steps: The melted slack wax is run into the sweating pan, which contains a small amount of hot water. This water is deep enough to cover a screen—usually made of one-quarter-inch mesh galvanized wire—which is held above the bottom of the pan by narrow strips of wood. Sweating pans are commonly about 20 feet long, 8 feet wide, and 8 to 10 inches deep, and are usually operated in units of 6 to 12 pans. After the desired amount of slack wax, usually from 4 to 6 inches, is run into the pan, it is cooled until the wax hardens into a solid cake; then the water is drawn off, and the cake of wax remains on the screen. The room containing the sweating pans (see Pl. II, *A*) is then heated very slowly.

The heat first melts out of the wax cake the constituents of lowest melting point and then, as the sweating proceeds, constituents of higher and higher melting points. As the sweating continues the melting point of the "stream" coming off the wax cakes is tested, cuts being made in the stream to divide the products off the pans into "foots oil" and "intermediate wax." When the wax left in the sweating pans has reached a satisfactory melting point and contains a predetermined minimum percentage of oil, sweating is discontinued. The wax thus obtained is treated with acid and filtered to the desired color. Plate III shows the wax storage and barreling house, with wax barreled for shipment.

TESTING METHODS IN PRESENT USE

Finished paraffin wax is valued commercially according to its melting point and its freedom from oil and other impurities. Tests for these properties in finished wax have been fairly well worked out.

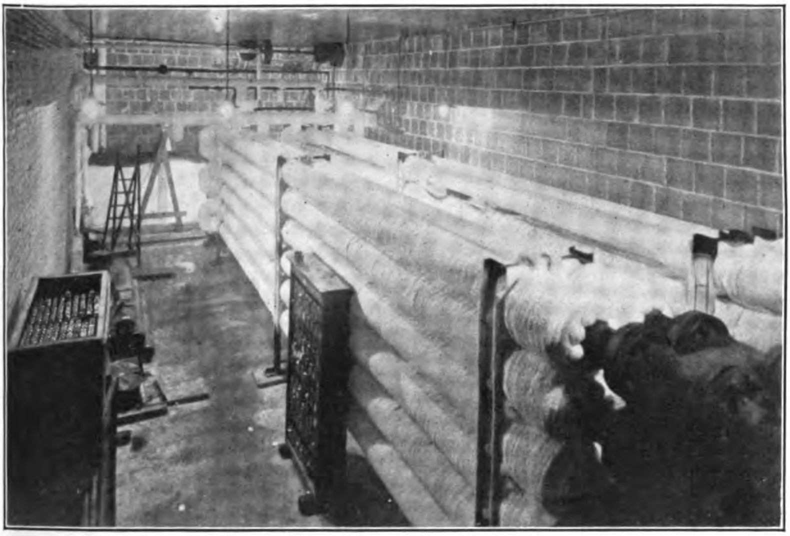
OIL CONTENT

The test that has been generally used to determine the oil content of wax is called the "press method."[7] This test is not satisfactory and its results are at best only comparative even for samples of relatively pure wax containing small amounts of oil. This method has been recommended and used only for testing commercial paraffin

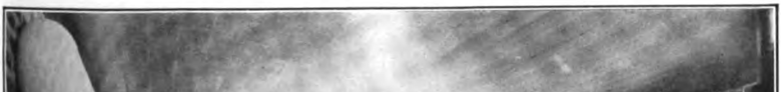
---

[6] Smith, A. D., work cited, pp. 373–399.

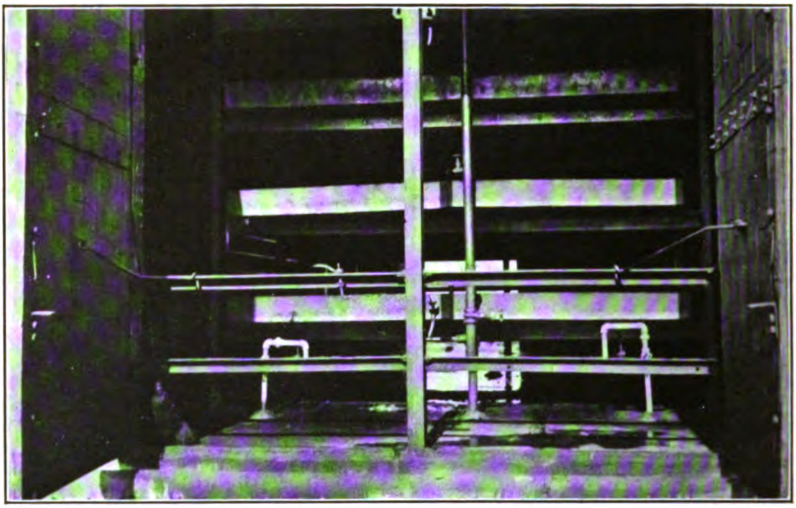
[7] Delbridge, T. G., Petroleum testing method; Chapter 7 of "A handbook of the petroleum industry," David T. Day, editor in chief. Vol. 1, 1922, pp. 724–725.



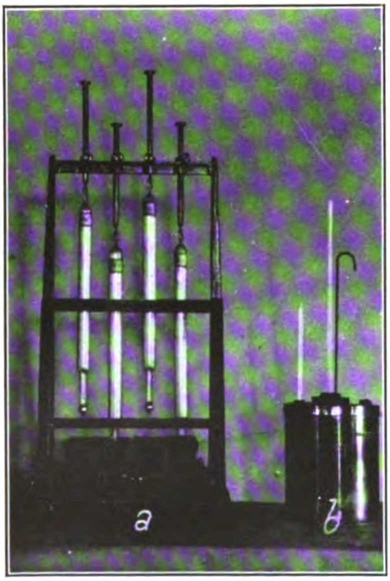
A. WAX-DISTILLATE CHILLING MACHINE



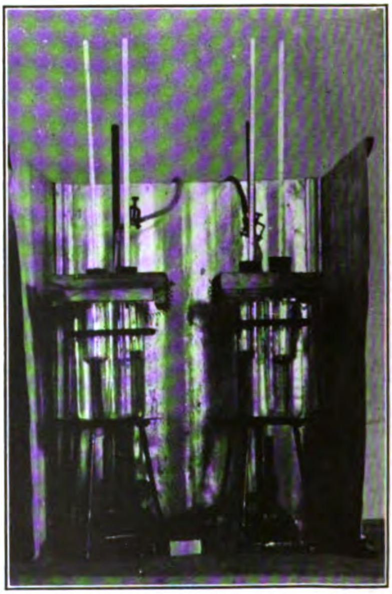
B. INTERIOR OF A PRESSROOM, SHOWING WAX PRESS AND ROOM-COOLING COILS



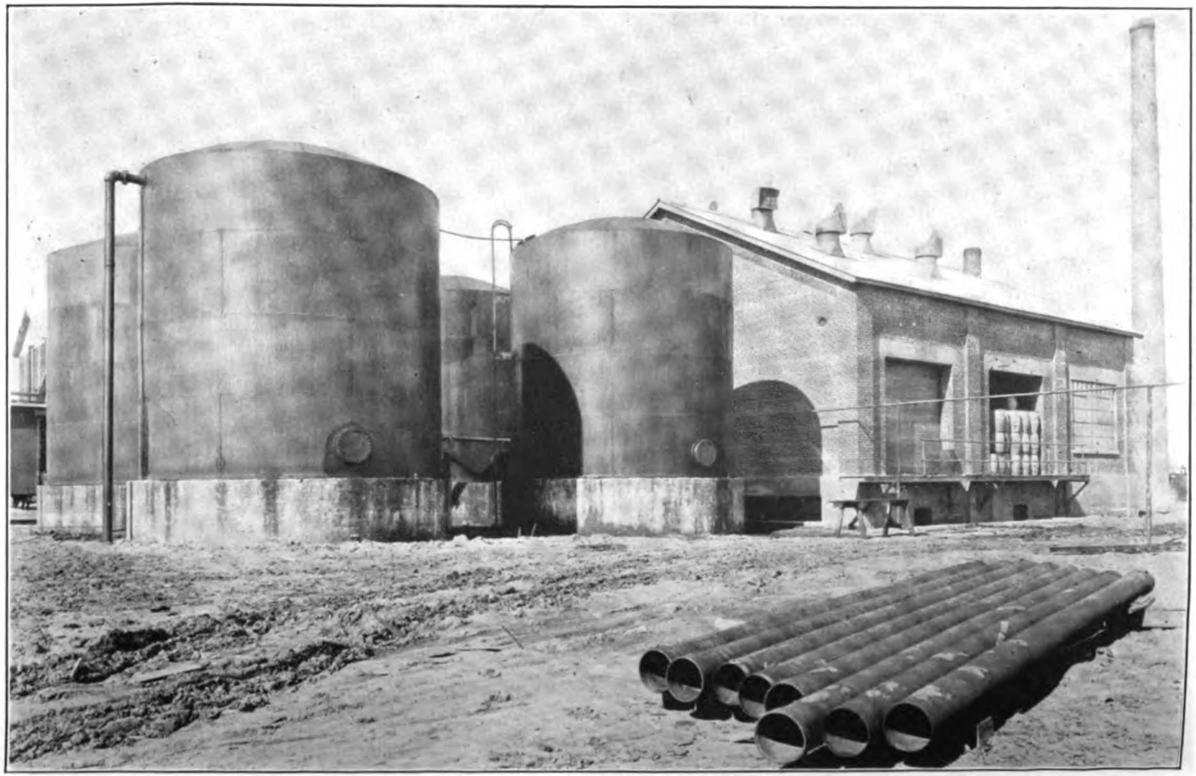
A. WAX-SWEATING HOUSE, SHOWING INTERIOR ARRANGEMENT OF SWEATING PANS



B. a, SAYBOLT IMPROVED WAX TESTER; *b*, A. S. T. M. PARAFFIN-WAX MELTING-POINT APPARATUS ASSEMBLY

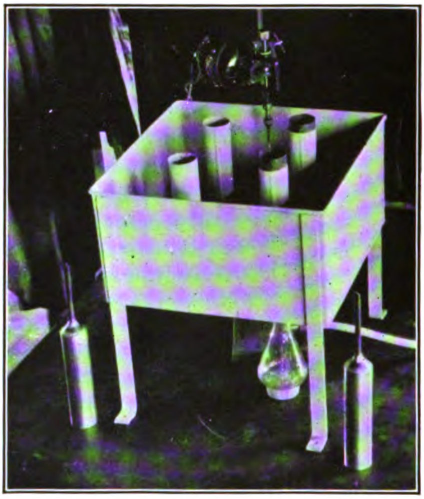


C. A. S. T. M. PETROLATUM MELTING-POINT APPARATUS ARRANGED TO RUN DUPLICATE TESTS

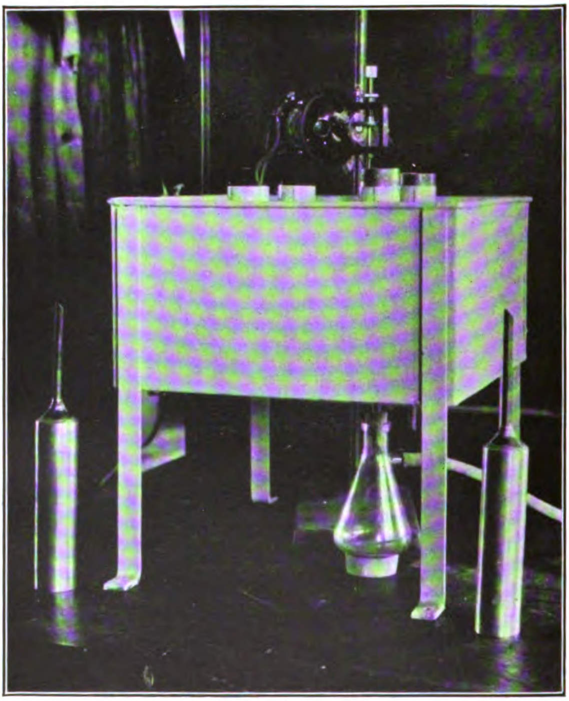


WAX STORAGE AND BARRELING HOUSE, CONTAINING BARRELED WAX READY FOR SHIPMENT

Original from UNIVERSITY OF MICHIGAN



A. TOP VIEW



B. SIDE VIEW

ASSEMBLY OF CONSTANT-TEMPERATURE FILTER-BATH ARRANGEMENT USED IN SEPARATING OIL-WAX MIXTURES WITH ACETONE

Original from UNIVERSITY OF MICHIGAN

that is practically free of oil. The nature of the test is such as to make it unsatisfactory for testing wax-oil mixtures that contain more than 10 per cent of oil. It is therefore useless for experimental work where the amount of oil may be 10 to 80 per cent of the sample.

MELTING POINT

The method generally used for measuring the melting point of a paraffin wax is in reality a method for measuring its crystallizing point, as the temperature recorded is that at which the latent heat of fusion is greatest. Two modifications of this method are described in Technical Paper 323 of the Bureau of Mines.[8] (See Pl. II, *B*.) One modification is generally known as the "Saybolt method," and involves using the well-known "Saybolt improved wax tester" shown at *a* in II, *B*. The other is the A. S. T. M.[9] paraffin-wax melting-point method shown at *b*. Plate II, *C*, shows the petrolatum melting-point apparatus arranged to run duplicate tests. Tests reported to the authors January 2, 1924, by E. B. Phillips, chief chemist, Sinclair Refining Co., Chicago, chairman of subcommittee III on paraffin wax of committee D-2 on petroleum products and lubricants of the American Society for Testing Materials, show that these two modifications check to 0.25° F.

AMERICAN SOCIETY FOR TESTING MATERIALS PARAFFIN-WAX MELTING-POINT METHOD

A volume of only 20 c. c. is needed for making a test by the American Society for Testing Materials paraffin melting-point method. The method is clean and convenient, and the rapidity of the temperature drop of the sample being tested is independent of the room temperature. The accuracy of this method for ordinary commercial waxes is remarkable, as two operators will often make perfect check runs.

Very thin copper tubes were tried in this test method and found as satisfactory as glass tubes. The temperature drop was faster, but the melting points did not differ more than 0.2° F. at most. Since there are several advantages in using these tubes, the use of the glass tubes has been discontinued unless the wax is found to be of very high oil content.

Table 2 gives the melting points and oil contents and Figure 1 the cooling curves of three typical commercial waxes (curves A, B, C) and of one intermediate wax (curve D). Study of these curves shows that as the percentage of oil increases the distinctiveness of the "melting point" disappears. Hence, although this method is satisfactory for use with good commercial waxes, it is not satisfactory for oil-wax mixtures. Plate II, *C*, shows the assembly of apparatus for this method.

---

[8] Federal Specifications Board, Specifications for petroleum products and methods of testing: Tech. Paper 323, Bureau of Mines, 1923, pp. 38–42.

[9] American Society for Testing Materials, A. S. T. M. standard adopted in 1922. 1922, pp. 24–27.

AMERICAN SOCIETY FOR TESTING MATERIALS PETROLATUM MELTING-POINT METHOD

Another method of determining melting points has recently been standardized by the American Society for Testing Materials for use particularly with noncrystallizable mixtures, such as petrolatum.[10-11] This method has the advantage of requiring only a small sample for test and of being applicable to all kinds of wax-oil mixtures, and it has therefore been used in the work described in this paper.

TABLE 2.—*Melting points and oil contents of four typical waxes*

Wax number	A. S. T. M. paraffin melting point	Oil content by acetone separation	Oil content by press method
	° F.	*Per cent*	*Per cent*
A	141.8	Nil.	0.30
B	128.2	3.5	.82
C	122.6	7.6	1.28
D	[a] 108.3	29.0	31.0

[a] Estimated; fell too rapidly to be an actual melting point.

For comparison, 80 tests of commercial quality paraffin waxes were run by both methods and it was found that the American Society for Testing Materials petrolatum method gave results that averaged 1.47° F. higher than the American Society for Testing Materials paraffin method. The maximum variation was only 3° F. in the 80 tests made. Plate II, *C*, shows the apparatus used in this method.

VALUE OF MELTING-POINT DETERMINATION OF OIL-WAX MIXTURES

At best, however, melting points do not and can not give reliable indications as to the composition of oil-wax mixtures. When three or more components are present several mixtures of different composition can be made which would have the same melting point. In the products made by sweating semirefined paraffin, particularly in the "foots oil" and "intermediate wax," the number of components is very large, consequently the melting point is of little value in indicating the composition of the mixture.

[10-11] American Society for Testing Materials, Tentative method of test for melting point of petrolatum: Proceedings, vol. 22, pt. 2, pp. 766–767. Federal Specifications Board, Specifications for petroleum products and methods for testing: Tech. Paper 323, Bureau of Mines, 1923, pp. 42–43.

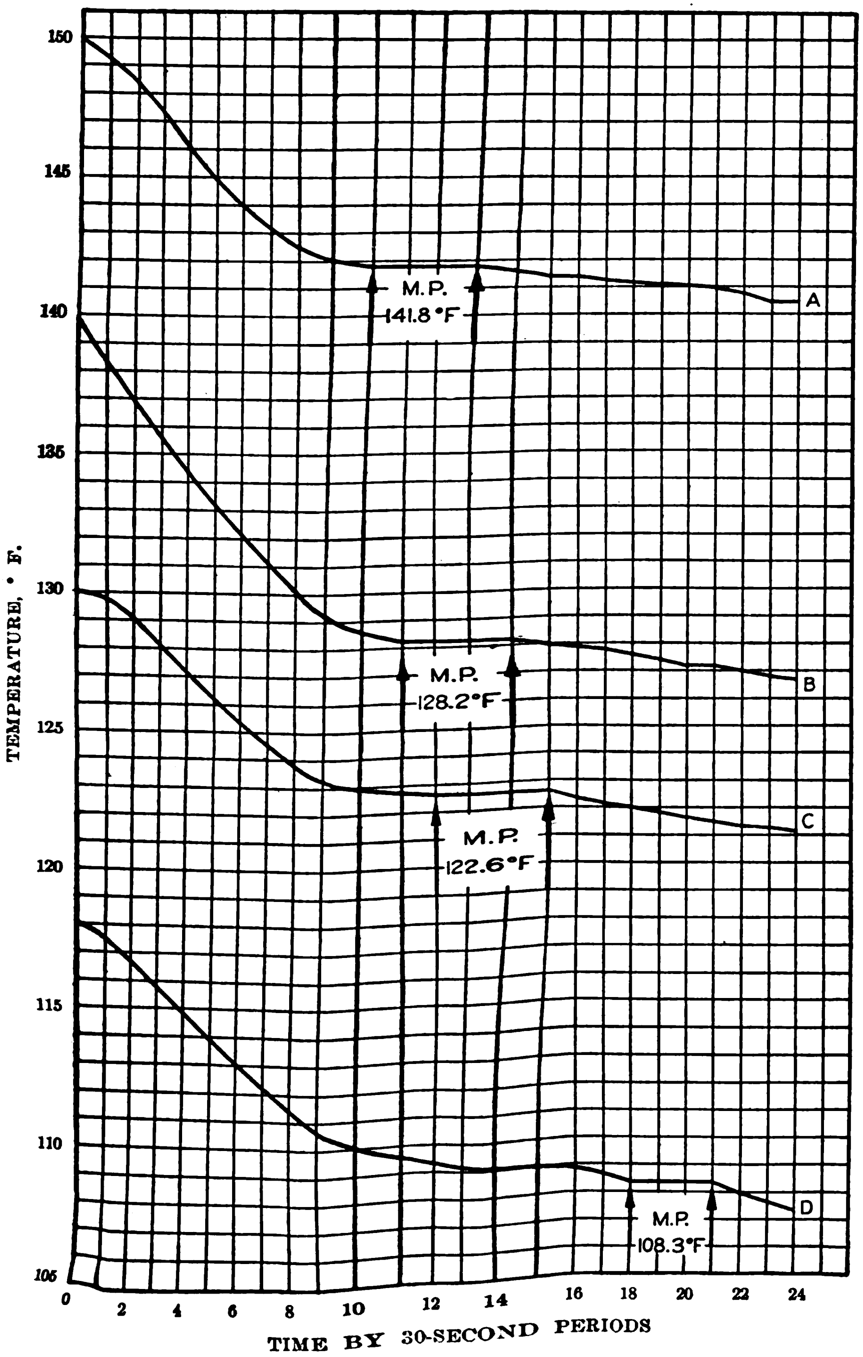


FIGURE 1.—Typical cooling curves of four typical waxes, A, B, C, and D (A. S. T. M. paraffin melting-point apparatus and method)

For example, a slack wax containing 60 per cent of 122° F. melting-point paraffin is divided by sweating into three fractions, foots oil, intermediate wax, and finished wax, with melting points of 60°, 110°, and 122° F., respectively. It might be assumed that all of the 122° F. paraffin would be contained in the finished wax. If this were true, the finished wax would amount to 60 per cent of the original slack wax. In practice, however, the finished wax is much less than this and it is therefore evident that part of the 122° paraffin has been dissolved during the sweating process by the lower melting products and will be found in the foots oil and intermediate wax.

In judging the efficiency of fractionation by sweating, one must know the amount of paraffin wax of a given melting point contained in the original slack wax and in the several sweated fractions or cuts made during the sweating run, therefore the melting points of the sweated cuts are of little value.

SPECIFIC GRAVITY

Since the difference in specific gravity between commercial wax, intermediate wax, and foots oil is very slight, even where these products are separated into relatively clear-cut fractions, it is evident that the specific gravity of a sweating cut is of practically no value for purposes of analysis or plant control when the cut contains many components in unknown amounts. For purposes of comparison, however, specific gravities, if desired, can best be determined at 130° F. by a specific gravity balance (Westphal).

WATER CONTENT

For determining the water content of a given sample, the Dean-Stark[12] method is used.

SOLUBILITY OF PARAFFIN WAXES AND OILS IN ACETONE

In all research and investigative work, direct methods are preferable to indirect, even though they frequently require more complicated apparatus and take more time than methods usually employed for routine work. In studying sweating, for example, a method by which the paraffin wax is actually isolated is preferable to any indirect method, such as melting-point or specific gravity determinations.

---

[12] Dean, E. W., and Stark, D. D., A convenient method for the determination of water in petroleum and other organic emulsions: Jour. Ind. and Eng. Chem., 1920, vol. 12, p. 486. Federal Specifications Board, Specifications for petroleum products and methods for testing: Tech. Paper 323, Bureau of Mines, 1923, pp. 65–66.

A reliable method for research work can be based on the selective solvent action of volatile organic compounds. The Holde[13] method of determining the paraffin-wax content of oil-wax mixtures is based on the selective solvent properties of a 50–50 mixture of alcohol and ether. This method is quite well known but has several distinctly undesirable features. The worst of these is the extremely low temperature at which precipitation, filtration, and washing are supposed to be accomplished. It has been found practically impossible to check results on the same sample closer than to 5 per cent of the wax content.

CHARACTERISTICS OF AN IDEAL SOLVENT FOR PARAFFIN OIL-WAX MIXTURES

For convenience and accuracy, a single organic compound is preferable to a mixture of compounds as a solvent. The following characteristics are necessary for a satisfactory solvent of oil-wax mixtures: The solvent must have a boiling point much lower than that of the oil or paraffin to be separated. It must have a comparatively high solvent power for oil and paraffin wax at elevated temperatures, and a low solvent power for paraffin wax and a comparatively high solvent power for oil at filtering temperatures. The physical condition of the paraffin wax thrown out of the solution must allow removal of the filtrate without removal of any of the precipitated paraffin wax. The solvent must be such that it can be easily and completely removed from the filtered paraffin wax.

SOLVENTS STUDIED

Very volatile gasoline, specific gravity 0.650 (86.2 A. P. I.), average boiling point 140° F., dissolved both wax and oil very readily. Upon cooling to 32° F., the paraffin settled out, apparently in a nearly pure state. With this solvent, however, it was difficult to keep the temperature at 32° F. during the process of separation and a slight increase of temperature dissolved out large amounts of paraffin. The precipitated wax was not easily pressed dry or easily washed; in fact it appeared to be an amorphous mass from which the gasoline could be separated only with difficulty.

Benzol gave somewhat better results than gasoline but the precipitate was again an amorphous mass rather than crystalline paraffin. Removal of the last traces of benzol from the paraffin was extremely difficult.

---

[13] Holde, D., The examination of hydrocarbon oils and of saponifiable fats and waxes. Translated by Edward Mueller, 1915, pp. 37–38.

Ethyl acetate gave good crystals of wax but there was particular difficulty in removing the last traces of this solvent from the crystallized portion of the paraffin.

ADVANTAGES OF ACETONE

Acetone seems to be especially adapted to the separation of paraffin wax and oil mixtures, as the following points of superiority over any other solvent tested indicate:

1. Acetone dissolves a large amount of either or both commercial paraffin wax and oil at temperatures near its boiling point. In fact, oils seem to be completely soluble in acetone at temperatures above 100° F.

2. Oil and low melting-point paraffins are still fairly soluble at temperatures of 60° to 80° F., while paraffin wax of commercial melting point is only slightly soluble at these same temperatures.

3. The separation of paraffin-wax precipitate from the solution of oil, paraffin wax, and acetone is peculiarly clear cut at temperatures below 90° F.

4. The crystalline structure of the precipitated wax is unique, as it can be filtered at considerable pressure without any of the paraffin wax being forced through the pores of the filter cloth. This is particularly noticeable if contrasted with the action of paraffin wax precipitated out of gasoline, benzol, or ether-alcohol solution.

5. Tests with chemically pure and U. S. P. acetone show practically no difference in the yield or character of wax, *provided the acetone is free from water.*

6. The solubility of commercial oil-free paraffin wax at temperatures between 50° and 70° F. is so small that any one temperature in this range can be selected as standard and uniform results can be obtained. This low solubility is particularly valuable, as it permits work to be done at or near ordinary room temperature.

7. The boiling point of acetone is 134° F., hence the solution can be easily evaporated on a steam bath. Acetone, moreover, is not as dangerous to handle in a laboratory as would be equal volumes of light gasoline or ether.

8. Only a few minutes time is required to remove the last traces of acetone from either paraffin or oil if the mixture is evaporated on a steam bath at 50 to 60 mm. pressure.

Because acetone was superior to any other solvent available, it was adopted for the present investigation and a detailed study made of its action as a selective solvent for oil and paraffin-wax mixtures.

QUALITY OF ACETONE USED

In this work U. S. P. acetone was used. To be sure that it did not contain water it was refluxed for 1 hour over anhydrous calcium chloride and then distilled; it gave a uniform product of 0.792 specific gravity at 60° F., b. p. 133.8° F. (56.6° C.) at 740 mm., these specifications fully conforming to those specified in general pharmaceutical practice as U. S. P. material. The used acetone was recovered from time to time by distillation, each lot of recovered acetone being tested for purity by taking the specific gravity before use.

PREPARATION OF OIL-FREE PARAFFIN WAXES

As commercial grades of paraffin wax usually contain some oil and low-melting wax, it was decided to prepare a set of waxes for use as standards. Several samples of paraffin wax were therefore purified by recrystallization from acetone until they were apparently free from all traces of oil. Figure 2 shows the changes in melting point of a typical wax in successive recrystallizations. In each crystallization wax was dissolved in approximately 10 volumes of hot acetone, the solution cooled to 80° F., and the crystallized wax removed by filtration. The acetone in the filtrate was recovered by distillation on a steam bath, the dissolved portion of the wax dried in vacuo, and the melting points of the precipitated and the dissolved waxes determined. As the reader will note, the melting points of both the precipitated and dissolved waxes increased rapidly at first, then more and more slowly, the increase becoming practically constant after the fourth crystallization. Although these melting points indicated that the crystallized wax was still a mixture of hydrocarbons, the wax was considered homogeneous enough for the present work.

The portion of the wax sample that was recovered from the filtrate looked yellow and oily in the first separation, only slightly yellow in the second, and almost entirely white in the third. In the third separation, and those thereafter, the wax had the general appearance and characteristics of ordinary paraffin wax of similar melting point. The crystallized portion did not change in color materially after the first separation, but when in a solid cake, it did appear more glazed and more nearly transparent as the melting point increased.

Several samples of oil-free wax were prepared in the manner just outlined. The original waxes had very different melting points and the final crystallized samples were expected to show similar differences. However, the samples of wax, after being freed from oil as described above, all had melting points of 122° F. or higher. Samples E–4, F–4, and G–4 (Table 3) are typical waxes thus prepared.

A low-melting sample of oil-free wax was prepared as follows: The filtrate from sample G–1 was cooled from 80° to 60° F. and filtered, yielding 33 grams of wax melting at 112° F. The filtrate from G–2 was similarly cooled and filtered, yielding 10 grams of wax melting at 111.3° F. These two samples were united and used as sample H, Table 3.

TABLE 3.—*Preparation of oil-free waxes*

Sample No.	Melting point (petrolatum)	Sample taken	Amount of acetone	Melting point of removed portion
		Grams	*C. c.*	*° F.*
E	129.7	300	3,000	98.1
E-1	131.7	253	2,530	112.4
E-2	133.9	229	2,290	117.2
E-3	134.7	215	2,150	119.7
E-4	135.0	207	(1)	(1)
F	119.2	450	3,000	105.7
F-1	121.3	410	2,700	107.6
F-2	121.4	379	2,500	108.0
F-3	122.5	353	2,330	108.0
F-4	122.2	336	(1)	(1)
G	110.1	450	4,000	(2)
G-1	116.0	324	3,240	90.0
G-2	121.4	254	2,540	101.0
G-3	122.3	247	2,470	106.7
G-4	123.5	202	(1)	(1)
H [3]	111.7	43	----------	----------

[1] Finished sample. [2] Very low. [3] Made from filtrates from G-1 and G-2.

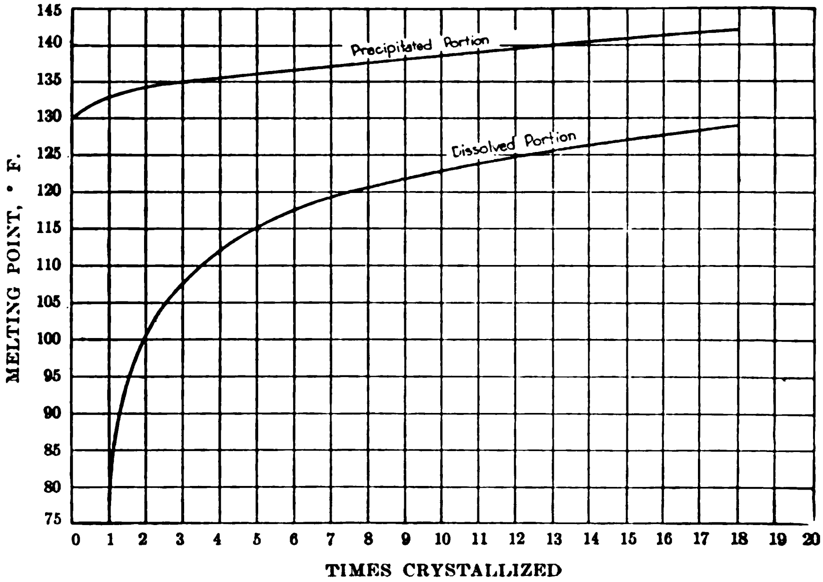


FIGURE 2.—Effect of recrystallization from acetone on the melting point of a typical paraffin wax

METHOD OF DETERMINING SOLUBILITY IN ACETONE

Preliminary experiments indicated that the method described in the following paragraphs could be used to determine the solubility of waxes and oils in acetone, and as it has proved reliable and convenient in practice it has been adopted as a standard in the present investigation.

A 10-gram sample of the oil or wax is dissolved by heating under a reflux condenser in enough acetone to make approximately 100 c. c. of solution. The solution is allowed to cool to a few degrees above the temperature at which the solubility is to be measured. During this cooling a part of the wax separates from the solution, either as crystals or as oily drops.

When waxes have a high melting point it is occasionally advisable to decant the solution from the separated wax some time before the desired temperature is reached because of the bulk of the crystals; occasionally a larger proportion of acetone may be required.

After cooling to nearly the desired temperature, the clear portion of the solution is decanted into a 4-ounce oil-sample bottle stoppered with a cork bearing an American Society for Testing Materials paraffin-wax melting-point thermometer and placed in a water bath. The bath is slowly cooled to the desired temperature and held there at least 30 minutes. The stopper containing the thermometer is now replaced by a two-hole stopper carrying a short glass tube connected to the air supply and a longer glass tube extending through the stopper and into the clear liquid about halfway between the surface of the solution and the precipitated wax. The upper end of this sampling tube is connected to a glass stopcocked U tube (Schwarts drying tube) into which 12 to 15 grams of the clear solution is transferred by very gentle air pressure. Both stopcocks are closed, the inlet tube rinsed with acetone and dried, and the U tube and contents weighed as quickly as possible. The weighed solution is quantitatively transferred to a tared flask and the acetone is evaporated on a steam bath. The residue is then dried, under a vacuum of 50 to 60 mm., to constant weight, and the solubility is calculated in percentage by weight of wax in acetone.

Figure 3 shows the set-up of the U tube and sample bottle used in making these solubility determinations. When work was being done at or above room temperature, the sampling tube extending into the liquid and the drying tube were heated as nearly as possible to the working temperature, otherwise the slight cooling of the clear solution caused precipitation that stopped up the sampling tube and its orifices.

This method of determining solubility was used to determine the influence of concentration of wax on the apparent solubility of wax in acetone; it was also used to determine the relations existing between the melting point of a wax and its solubility at different temperatures in acetone.

EFFECT OF WAX CONCENTRATION ON SOLUBILITY

Table 4 shows the effect of varying the size of the wax sample taken for a solubility determination. Samples of wax E–4 (Table 3), weighing 15, 10, and 8 grams, respectively, were each dissolved in acetone to make 100 c. c. volume and the solubilities determined at four temperatures. Each figure shown is the average of three determinations.

TABLE 4.—*Effect of wax concentration on solubility*

[Solubilities in per cent by weight of wax in acetone]

Temperature	15-gram sample	10-gram sample	8-gram sample	Maximum difference
°F.				
120	8.59	6.92	6.32	2.27
110	3.76	3.17	3.17	.59
100	1.57	1.56	1.55	.02
90	.753	.751	.750	.003
70	.201	.200	.200	.001

The reader will note that the influence of concentration is quite important at high temperatures, but below 110° F. the difference in solubility becomes very small and is negligible at 90° F. and below. The difference in apparent solubility at high temperature is probably due to the presence in the sample of relatively low-melting material which is more soluble at high temperatures than high-melting wax.

RELATION BETWEEN MELTING POINTS AND SOLUBILITY

The probable accuracy of the method of determining solubility as well as the actual solubilities of typical oil-free waxes are shown in Table 5. In general the method is most reliable when employed at temperatures from 60° to 90° F. At higher temperatures the volatility of the acetone and at lower temperatures the light weight of the dissolved wax make the results less certain. It is evident, however, that the solvent power of acetone for all grades of wax decreases rapidly as the temperature is lowered and that there is much less difference between the solubilities of different grades of wax at low than at high temperatures. These facts are also brought out by the solubility curves in Figure 4. The physical properties and methods of preparing the waxes used in determining the solubilities given in Table 5 and shown graphically in Figure 4 are shown in Table 3.

PREPARATION OF WAX-FREE OILS

Commercial slack waxes were dissolved in acetone in the approximate ratio of 10 grams of sample to 100 c. c. of acetone, cooled to 60° F., and fitered. The dissolved oily portion of each slack wax was recovered by distilling off the major portion of the acetone and then removing the last traces of the solvent in vacuo. Obviously these oil portions still contain some paraffin wax. To find out the quality and quantity of the remaining wax a sample of each oil was tested as follows: A 5-gram sample was dissolved in 50 c. c. of acetone, cooled to 50° F., and filtered, then the wax precipitate was washed with 50 c. c. of cold (32° F.) acetone. The precipitate was dried in vacuo, weighed, and its melting point taken. The results are shown in Table 6.

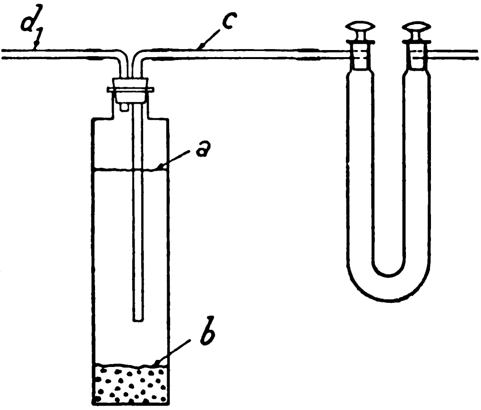


FIGURE 3.—Detail of set-up of U tube and sample bottle used in making solubility determinations: *a*, Clear liquid level; *b*, crystallized-wax level; *c*, sampling tube connected to U tube; *d*, tube for applying air pressure.

TABLE 5.—*Solubility of oil-free paraffin wax in acetone*

[Percentages by weight]

Temperature	Wax E-4				Wax G-4				Wax H			
	Determination			Average	Determination			Average	Determination			Average
	1	2	3		1	2	3		1	2	3	
°*F.*												
120	6.85	7.00	6.92	6.92	11.39	11.40	11.38	11.39	------	------	------	------
110	3.16	3.15	3.20	3.17	5.68	5.67	5.68	5.68	12.69	12.69	12.62	12.67
100	1.55	1.55	1.55	1.55	2.48	2.53	2.53	2.51	8.85	8.82	8.82	8.83
90	.750	.751	.749	.750	1.09	1.10	1.08	1.09	3.26	3.25	3.30	3.27
80	.369	.358	.325	.350	.733	.736	.698	.722	1.59	1.59	1.54	1.54
70	.200	.210	.190	.190	.385	.387	.392	.388	.752	.715	.704	.723
60	.125	.124	.126	.125	.183	.182	.182	.182	.343	.339	.287	.323
50	.054	.069	.062	.061	.088	.082	.081	.083	.150	.149	.149	.149
32	.034	.031	.021	.028	.046	.039	.037	.040	.053	.055	.057	.055

TABLE 6.—*Tests of wax-free oils*

Sample No.	Wax removed at 60° to 50 °F.	Melting point of wax	Pour point of remaining oil
	Per cent	°*F.*	°*F.*
I	5.35	88.0	45
J	4.40	99.7	55
K	5.20	90.8	45
L	3.72	100.9	45
M	3.80	97.1	50
N	4.50	95.7	50
O	5.85	95.1	50

Another sample, P, was prepared in the same manner as those shown in Table 6, except that the filtration was made at 29° F. instead of 60° F. The cold test of this oil was 30° F.

These tests show that the amount of wax remaining in each of these oils is small, has a very low melting point, and can not be considered commercial wax.

Solubility determinations were made with four of the above samples of oil (I, J, K, and P), and low-melting-point wax mixtures. The results of these determinations are shown in Table 7 and graphically represented in Figure 5.

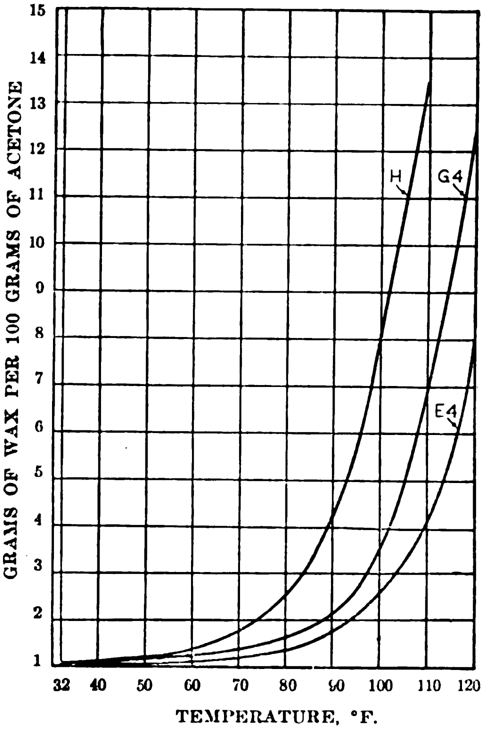


FIGURE 4.—Solubility curves of three oil-free waxes, E4, G4, and H

TABLE 7.—*Solubility of wax-free oils in acetone*

[Percentage by weight]

Temperature	Oil I				Oil J			
	Determination			Average	Determination			Average
	1	2	3		1	2	3	
°F								
95	22.64	22.85	22.94	22.81	30.97	31.05	31.04	31.02
80	12.09	12.05	12.01	12.05	16.30	16.24	16.31	16.28
70	8.15	8.49	8.44	8.48	10.45	10.43	10.41	10.43
60	4.250	4.403	4.328	4.327	6.585	6.600	6.618	6.601
50	1.903	1.875	1.908	1.895	3.392	3.309	3.414	3.398
32	0.489	0.463	0.491	0.481	1.488	1.482	1.464	1.478

Temperature	Oil K				Oil P			
	Determination			Average	Determination			Average
	1	2	3		1	2	3	
°F								
95	55.15	55.06	55.69	55.30	----------	----------	----------	----------
80	18.77	18.80	18.32	18.63	37.04	36.90	36.90	36.95
70	12.25	12.20	12.29	12.24	21.87	21.84	21.83	21.83
60	7.377	7.485	7.520	7.461	14.27	14.32	14.36	14.32
50	4.147	4.140	4.207	4.198	9.562	9.566	9.656	9.594
32	1.810	1.824	1.816	1.817	5.761	5.741	5.731	5.744

METHOD OF DETERMINING THE PARAFFIN-WAX CONTENT OF OIL-WAX MIXTURES

INTERSOLUBILITY OF OIL AND WAX IN ACETONE

Comparison of Figures 4 and 5 indicates that there is a real difference between the solubility in acetone of paraffin wax and the oil that must be removed from it in the refining process. However, the fact that oil and paraffin wax are both soluble in acetone and in each other must be considered when any method using this solvent is standardized.

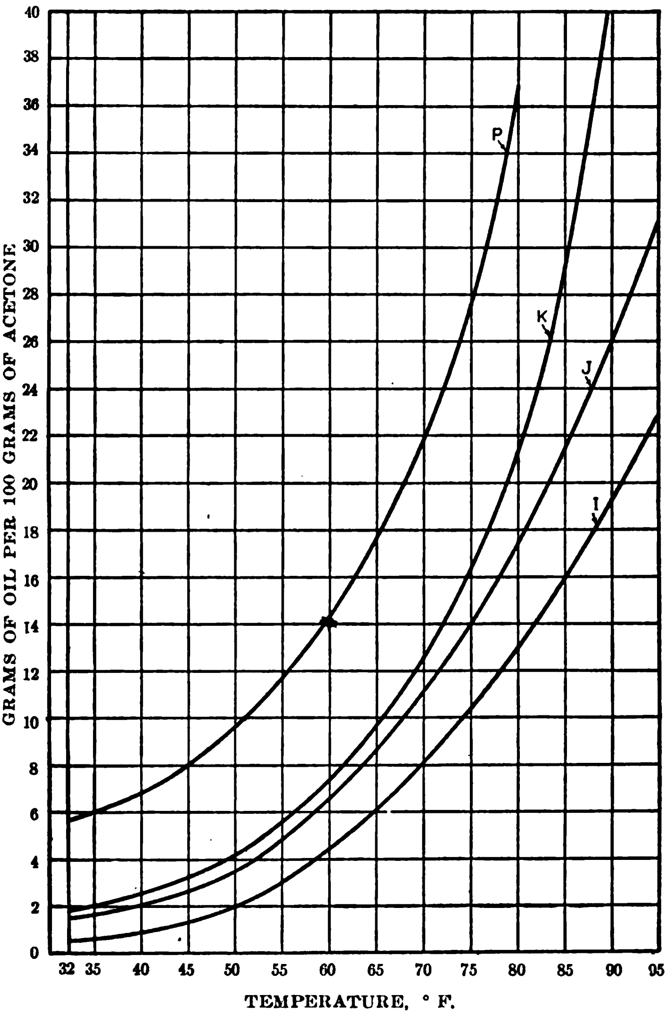


FIGURE 5.—Solubility curves of four wax-free oils

To determine the extent of this intersolubility would require an elaborate investigation which is not within the scope of this report. Tests were made, however, of several mixtures of various waxes and oils, and it was found that the intersolubility became greater as the oil and paraffin wax diverged in character. For example, a low-pour-point oil (sample P, Table 7) had a much greater effect on the solubility of high-melting-point paraffin wax than did samples I, J, or K (45° to 55° F. pour point). In general, it was found that the intersolubility in acetone of oil and wax in a normal "slack wax" was very slight. Since the present investigation deals only with such mixtures and especially since the details of the proposed method are carefully specified, this consideration can be neglected.

WAX CONCENTRATION

As the solubility of paraffin wax in acetone is appreciable at 32° F. or above, the smallest amount of acetone necessary to remove the oil should evidently be used. It is unsatisfactory, however, to attempt to vary the amount of solvent used for different oil-wax mixtures so that the relation between the amount of oil to be removed and the acetone used will remain constant. For example, a sample that consists mainly of oil is much more soluble in acetone than one that consists mainly of wax; consequently, for complete solution a much greater ratio of acetone to oil is required for high-wax mixtures. Therefore, for the routine testing of oil-wax mixtures of different composition an arbitrary standard method based on the relative solubilities of oil and wax in acetone is necessary.

The effects of varying the amount of solvent and of various methods of precipitating and washing the crystallized paraffin were studied by making 18 trial determinations as follows: Three samples of slack wax, Q, R, and S, whose approximate oil and wax content were known, were selected and treated by the following methods:

METHOD 1

The sample was dissolved in acetone by refluxing and was cooled to 68° F. (20° C.), then filtered through a fine linen cloth and pressed as dry as possible by hand. The crystallized wax portion was folded in the linen cloth and the pack thus made was folded in heavy canvas and pressed dry between two oak blocks in an ordinary screw vise. The wax cake was freed from traces of acetone by evacuating over a steam bath. The wax was then filtered hot through filter paper to remove possible dust and lint.

METHOD 2

The same procedure was followed as above except that the dry wax cake as taken from the press was finely broken up and washed for 10 minutes in cold acetone, with a mechanical stirrer. The amount of acetone for washing was taken arbitrarily as one-half of the amount previously used for solution. After it had been washed, the wax was filtered and pressed dry as in method 1. The wax was freed from remaining traces of acetone and filtered.

METHOD 3

The same procedure was used as in method 1, except that the wax portion obtained was crystallized from fresh acetone a second time, pressed, freed from acetone, and filtered.

APPLICATION OF METHODS

Method 1 was applied to all three waxes and the amount of acetone used for solution was varied in different tests so the amount of oil was respectively 10, 8, 6, and 4 per cent of the weight of acetone.

Methods 2 and 3 were applied to slack wax Q at the rate of 10 per cent of oil to the acetone used for solution and to slack wax R at the 8 and 6 per cent rates.

The wax portions thus obtained were submitted to two refinery laboratories for determination of the oil content by the press method. On account of the well-known limitations of this method, these two laboratories differed considerably in the percentages of oil they reported. The set of determinations which in general were the highest was selected.

Table 8 shows the data obtained for the 18 samples. Since the maximum oil content allowed for crude scale waxes is usually 2 per cent, and for finished commercial wax less than 1 per cent, as determined by the press method, some relative idea of the freedom of these samples from oil can be gained.

These tests show that if only one crystallization is used the oil to be removed can not be more than about 5 per cent of the acetone used. If the crystallized wax is thoroughly washed with new acetone or crystallized a second time from new acetone, the amount of oil to be removed can be as much as 15 per cent of the solvent.

Further recrystallization showed that the recrystallized samples were practically free of oil, and the amount of "oil" shown by the press method is undoubtedly high.

Attention is called to the low melting point of all samples of wax obtained from slack wax R. This slack wax was selected because its characteristic wax had a very low melting point, only 122° F., when free of oil.

TABLE 8.—*Effects of varying the concentration and method of treating slack waxes*

Slack wax No.	Sample No.	Oil based on weight of acetone	Acetone used	Acetone for wash	Acetone used for second crystallization	Wax sample	Wax recovery	Melting point of recovered wax	Oil by press method in recovered wax
		Per cent	*C. c.*	*C. c.*	*C. c.*	*Grams*	*Per cent*	° *F.*	*Per cent*
Q	1	10	1558			200	47.85	121.4	6.14
Q	2	8	2150			200	41.65	123.8	1.13
Q	3	6	2562			200	40.15	124.0	.29
Q	4	4	3875			200	36.75	126.2	.45
Q	5	10	1558	780		200	41.10	125.1	.87
Q	6	10	1558		1020	200	37.28	125.2	.38
R	1	10	682			150	66.60	119.0	2.22
R	2	8	853			150	65.25	120.0	1.96
R	3	6	1138			150	63.65	122.1	1.14
R	4	4	1720			150	59.00	122.0	1.29
R	5	8	1705	876		300	57.50	118.3	.51
R	6	8	1705		1134	300	49.60	122.5	.51
R	7	6	2276	1138		300	53.33	119.1	.68
R	8	6	2276		2064	300	52.23	122.5	.20
S	1	10	823			150	58.16	122.6	3.35
S	2	8	1028			150	56.16	124.0	3.31
S	3	6	1368			150	54.75	125.1	2.42
S	4	4	2027			150	47.41	126.0	.32

Since 10 grams of oil can be removed with 100 grams of acetone, if the wax crystals are well washed, and furthermore, since 10 grams of high-melting-point commercial paraffin is about all that 100 grams of boiling acetone will dissolve, this concentration would seem to be well adapted for this work. However, since 100 grams of acetone is approximately 135 c. c., it was decided to try using 10 grams of sample and 100 c. c. of solvent for solution. This procedure has worked out most satisfactorily and since it is more convenient has been adopted for this work.

TEMPERATURE OF FILTRATION

The filtrations for the data shown in Table 8 were made at 68° F. (20° C.), but an appreciable amount of dissolved wax was carried through with the filtrate at this temperature. Trial runs with various oil-wax mixtures showed that the most satisfactory and convenient temperature was 60° F. Although it is true that paraffin wax is slightly soluble in acetone at that temperature (see fig. 4, p. 16), it is also true that at lower temperatures the solubility of acetone for oil is much decreased (see fig. 5, p. 17). The wax remaining in solution at 60° F. (using 10 grams of sample to 100 grams of acetone) has a very low melting point, as shown by the data in Table 6. Subsequent work, when 60° F. was used as a standard temperature for filtration, shows that the error introduced must be less than other unavoidable compensating errors, as excellent check determinations can be made. Other advantages of 60° F. as a standard temperature are: First, it is not removed so far from average room temperature; second, it has long been used as a basic temperature in the oil industry.

STANDARDIZING EQUIPMENT

After much investigation, a filtering bath and filters, as shown in detail in Figure 6 and Plate IV, *A* and *B* (p. 5), were developed. The water bath and tubes *a* are made of heavy galvanized iron. The four filters are made of brass tubing with three-eighths-inch copper tubing for the outlet *f*. A Monel metal screen (very fine mesh) is placed across the filter, as shown at *e*. The cut-away section of the elevation shows the manner in which the filters are assembled in the bath. A cork, *c*, is placed under the filter and a Gooch rubber is placed around the top at *g*, to prevent any air circulation around the filter. Several test-tube holders, *b*, were placed around two sides of the bath as shown.

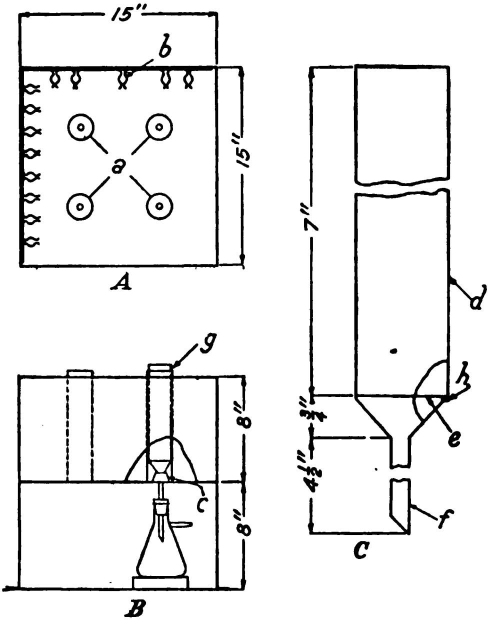


FIGURE 6.—Filtering bath and filters: *A*, Plain view; *B*, elevation; *C*, detail of filter tube; *a*, tubes; *b*, test-tube holders; *c*, cork; *d*, filter tube; *e*, position of monel-metal screen; *f*, outlet; *g*, position of Gooch rubber; *h*, soldered joint

TESTING FOR WAX CONTENT OF OIL-WAX MIXTURES

SAMPLING OIL-WAX MIXTURES

It is extremely important in testing oil-wax mixtures that the sample obtained be truly representative. This is not easily done unless the mixture to be sampled is melted, thoroughly stirred, and a sample immediately drawn while hot. If the mixture is allowed to cool to a solid or semisolid cake the contained wax segregates and to obtain a representative sample by cutting out part of the cake is practically impossible.

When accurate results are desired the sample should be filtered hot through filter paper, as the presence of foreign matter not only makes the weighing inaccurate but may cause the precipitate to form in hard masses and thereby prevent complete washing.

PROCEDURE IN TESTS

The test procedure is as follows: A 5-gram sample is weighed into a 100-c. c. Erlenmeyer flask; 35 c. c. of acetone is refluxed with the sample for a few minutes, and the solution is transferred while hot to a 12-inch 120-c. c. capacity test tube, which is immediately stoppered to prevent evaporation; 15 c. c. of acetone is then used to wash out the flask and added to the other solution, thus making 5 grams of sample and 50 c. c. of acetone. The solution is shaken vigorously—in cool water if desired—until it has cooled to nearly 60° F., and then placed in the bath and allowed to become adjusted to the bath temperature of 60° F.

Test tubes each containing 50 c. c. of new acetone are also kept at 60° F. in the bath. It has proved convenient to have these tubes graduated in one 20-c. c. portion and two 15-c. c. portions.

When the solution has had ample time to come to the bath temperature, it is poured into the filter and the acetone solution is allowed to drain off. Then 15 c. c. of the cool acetone is put into the tube that originally contained the sample. The tube is shaken quickly and the contents poured into the filter. A 15-c. c. volume is again poured into the sample tube, shaken, and poured into the filter. After each addition of wash acetone the filter is allowed to drain. The 20-c. c. volume of wash acetone remaining is then used to wash down any crystals remaining on the sides of the filter. As soon as this last volume is added the suction is attached and the filter is allowed to drain dry, which requires about 30 seconds.

The filtrate is distilled over a steam bath until practically all the acetone is recovered. The oil portion is then quantitatively transferred to a small Erlenmeyer flask, distilled, and dried in vacuo in a boiling-water bath. The oil is weighed and the wax calculated by difference. The wax precipitate is recovered from the filter and evacuated in a boiling-water bath, no effort being made to make a quantitative weighing of it. Its melting point can then be determined.

Experience with the above method has shown that it generally gives reliable results if the melting point of the wax portion is normal. If the melting point of the recovered wax is low the recovered wax is redissolved with new acetone at the rate of 20 c. c. of acetone to 1 gram of wax. The solution is cooled to 60° F., filtered, and the wax crystals washed with 20 c. c. of cool acetone per gram of sample. The oil portion thus removed is recovered as above outlined by distilling the filtrate and evacuating to remove the last trace of acetone. The wax portion is likewise evacuated and its melting point determined. The total amount of oil removed is then determined and the yield of wax thus found by difference. If, on the other hand, the melting point of the recovered wax is abnormally high the percentage yield will probably be slightly low and for accurate results it is necessary to recover an additional crop of wax crystals from the separated oil by redissolving in acetone, cooling to 60° F., and filtering as above. The melting point of the combined wax portions recovered from the sample being analyzed is then determined.

This procedure gives a good clean wax and very consistent results. Two points, however, must be carefully watched: First, the solution must be shaken while cooling until it is nearly at the filtering temperature, so that the wax precipitating out will not form a lumpy mass which is not easily washed; second, care must be taken that no water gets into the sample during the operation, as a small amount of water greatly decreases the solvent power of the acetone for both oil and wax. An attempt to use suction during the entire operation of filtering was not successful, because the temperature of the filter, the contained solution, and the wax became too low, owing to the rapid evaporation of the solvent.

APPLICATION OF METHOD

The method of analysis just described has been successfully used for analyzing slack waxes, or such oil-wax mixtures as are obtained when slack wax is sweated to yellow scale or finished paraffins. For example, two slack waxes, T and U, were treated with acetone in two crystallizations in the manner just outlined. Slack wax T yielded 53.64 per cent of wax, M. P. 122.0° F.; slack wax U yielded 50.73 per cent of wax, M. P. 126.4° F.

TABLE 9.—*Determination of the distribution of paraffin wax in the sweated fractions of a typical slack wax*

Cut No.	Slack wax	First crystallization			Second crystallization			Total wax	M. P. of total wax
		Wax	Sample	M. P. of wax	Wax	Cut	Sample		
	Per cent	*Per cent*	*Per cent*	°*F*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	°*F.*
1-T	7.92	36.57	2.90	97.9	37.10	13.57	1.07	1.07	116.6
2-T	13.22	41.02	5.42	105.1	53.88	22.10	2.92	2.92	116.3
3-T	19.36	42.12	8.15	105.7	56.73	23.89	4.62	4.62	115.5
4-T	7.97	44.91	3.58	110.0	73.02	32.79	2.61	2.61	116.9
5-T	0.98	60.74	0.60	114.7	76.08	47.21	0.46	0.46	118.8
6-T	0.69	66.23	0.46	114.6	78.04	51.69	0.36	0.36	118.7
7-T	2.96	75.61	2.24	116.4	79.19	59.88	1.77	1.77	119.3
8-T	4.23	82.65	3.50	117.1	81.27	67.17	2.84	2.84	120.5
9-T	7.97	86.44	6.89	117.6	83.50	72.18	5.75	5.75	122.0
10-T	33.25	90.54	30.10	124.5	58.75	5.55	1.85	31.95	124.5
Total	99.55		63.84					54.35	121.0
1-U	1.44	39.25	0.57	102.9	50.72	19.90	0.29	.29	119.5
2-U	2.91	43.05	1.25	112.3	63.06	27.15	0.79	.79	121.9
3-U	5.45	43.10	2.35	113.6	67.23	28.98	1.58	1.58	122.7
4-U	10.32	44.63	4.61	115.5	67.89	30.30	3.13	3.13	123.3
5-U	13.86	42.14	5.84	117.8	73.06	30.79	4.27	4.27	123.6
6-U	16.30	43.55	7.10	118.6	74.73	32.55	5.31	5.31	123.8
7-U	24.76	48.64	12.04	122.1	1.52	0.78	0.19	12.23	122.1
8-U	4.23	65.50	2.77	127.0	2.73	0.94	0.04	2.81	127.0
9-U	4.55	73.32	3.34	128.5	3.56	0.95	0.04	3.38	128.5
10-U	14.60	89.21	13.02	134.3	21.21	2.29	0.33	13.35	134.0
Total	98.42		52.89					47.14	126.9

These slack waxes were charged to a semicommercial sweating oven and the temperature of the oven gradually increased, the wax being thus sweated down very gradually, just as it would be in a commercial wax plant. The samples sweated off were collected and removed at regular intervals of temperature and time, in such a manner as to give nine fractions of foots oil and intermediate wax and one sample (remaining in the sweating pans) of finished paraffin wax. These fractions were numbered respectively 1–T to 10–T and 1–U to 10–U. The second column in Table 9 shows the percentage of the sweating sample taken off as in various fractions.

The sweated fractions were then each analyzed for their wax content by the standard method (5-gram sample to 50 c. c. of solvent for solution and washing with 50 c. c. at 60° F.) outlined above. The wax yield of each fraction by difference is shown in the third column of the table. The melting point of each wax thus separated is shown in column 5.

Upon the completion of the analysis of each set it was found that the total percentage of wax thus recovered exceeded the amount which the preceding analysis had shown the respective slack waxes to contain, and the melting points of most of the wax portions were low. The wax and oil portions of the fractions were therefore recrystallized, with the results shown in Table 9 under " Second crystallization." In this work the melting points of the waxes recovered from the original slack waxes were taken as normal, namely 122.0° F. for sample T and 126.4° F. for sample U.

The total percentages of wax thus obtained were 54.35 per cent and 47.14 per cent, respectively. The percentages were recalculated to compensate for the handling loss in sweating, becoming 54.6 per cent for all fractions of T and 48.5 per cent for all fractions of U. All the wax fractions of each set were combined and the melting point of all wax for each set was taken.

For comparison the results of the original analyses and the net results of the analyses of the sweated fractions are tabulated below as Table 10.

TABLE 10.—*Comparison of analysis of original slack wax with the analysis of its sweated fractions*

Slack wax sample	By slack wax analysis		By separate analysis of fractions		Difference	
	Per cent	M. P.	Per cent	M. P.	Per cent	M. P.
T	53.64	122.0	54.60	121.0	+0.96	−1.0
U	50.73	126.4	48.50	126.9	−2.23	+0.5

Digitized by Google Original from UNIVERSITY OF MICHIGAN

The method outlined above is admittedly long when applied to many fractions, as are obtained when a given slack wax is being closely analyzed for detailed information. The method has been used with numerous samples, however, and found extremely useful, for the results were always quite close. In every test the yield of wax has been compatible with the melting point, thus making results particularly valuable for comparative purposes. No effort has been made to standardize the solvent method outlined above for use in determining the oil content of commercial paraffin wax. It has been found useful as a method of analysis of oil-wax mixtures for research and plant control.

SUMMARY

This paper reviews the methods now in general use for testing oil-wax mixtures and describes a new method, which depends on the relative insolubility of paraffin wax in acetone for use in determining the oil content of oil-wax mixtures.

The American Society for Testing Materials, paraffin-wax melting-point method has been found an accurate and satisfactory method of determining the melting point of commercial paraffin-wax or wax-oil mixtures of relatively low oil content; however, the method is unsatisfactory when the oil content is as much as 20 per cent.

The most satisfactory method of determining the melting point of oil-wax mixtures, in which the oil content is 15 per cent or more, is the American Society for Testing Materials, petrolatum melting-point method.

The various experiments made in developing the use of acetone as a selective solvent for oil-wax mixtures showed the following interesting facts:

The difference in solubility of commercial grade oil-free paraffin wax in acetone between 50° and 70° F. is only 0.3 per cent. For high-melting-point oil-free paraffin it is only 0.13 per cent, thus making any one temperature between these limits satisfactory for comparative testing.

The quality or melting point of the paraffin affects the solubility more than does an increase of temperature between 50° and 70° F.

The amount of oil soluble in acetone at 60° F. is from 5 to 15 per cent, depending upon the nature of the oil. This solubility decreases until it is only 2 to 10 per cent when the temperature is reduced to 50° F., making 60° F. a more suitable working temperature for separation of oil-wax mixtures than lower temperatures.

If the wax portion of a sample is crystallized out of acetone a second time, it is more nearly free of oil than any ordinary commercial paraffin wax.

The matter of obtaining a truly representative sample of an oil-wax mixture is very important.

In testing oil-wax mixtures for their wax content, the wax portion has to be crystallized out of acetone a second time if its melting point is abnormally low. If the melting point of the wax portion is abnormally high the best procedure is to recover an additional crop of wax crystals from the oil portion of the separated sample by crystallizing it out of acetone a second time.

The accuracy of the above method of analysis increases as the percentage of wax contained in the sample or the melting point of the wax increases.

○